



June 15, 2016

Tom Moe USS Corporation P.O. Box 417 8771 Park Ridge Dr Mountain Iron, MN 55768

RE: Project: NPDES-TB Wk1 Pace Project No.: 1267255

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on June 01, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Melisa M Woods

Massia Wirds

melisa.woods@pacelabs.com

Project Manager

Enclosures

cc: Cory Hertling Terri Sabetti, NTS



315 Chestnut Street Virginia, MN 55792 (218) 742-1042



CERTIFICATIONS

Project: NPDES-TB Wk1
Pace Project No.: 1267255

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792

Alaska Certification #MN01084

Arizona Department of Health Certification #AZ0785

Minnesota Dept of Health Certification #: 027-137-445

North Dakota Certification: # R-203

Wisconsin DNR Certification #: 998027470

WA Department of Ecology Lab ID# C1007

Nevada DNR #MN010842015-1

Oklahoma Department of Environmental Quality

Duluth Minnesota Cerification ID's

4730 Oneota St., Duluth, MN 55807

Minnesota Dept of Health Certification #: 027-137-152

Wisconsin DNR Certification #: 999446800

North Dakota Certification #: R-105





SAMPLE SUMMARY

Project: NPDES-TB Wk1

Pace Project No.: 1267255

Lab ID	Sample ID	Matrix	Date Collected	Date Received
1267255001	SD 001 (Seep 020)	Water	06/01/16 11:00	06/01/16 14:25





SAMPLE ANALYTE COUNT

Project: NPDES-TB Wk1

Pace Project No.: 1267255

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
1267255001	SD 001 (Seep 020)	EPA 1664 TPH	BT1	1	PASI-DUL
		USGS I-3765	BEM	1	PASI-V
		EPA 300.0	DMB	1	PASI-V



ANALYTICAL RESULTS

Project: NPDES-TB Wk1

Pace Project No.: 1267255

Date: 06/15/2016 04:51 PM

Sample: SD 001 (Seep 020)	Lab ID:	Lab ID: 1267255001		Collected: 06/01/16 11:00		Received: 06/01/16 14:25 Matrix: Water			•
Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
1664 SGT-HEM, TPH	Analytical	Method: EPA	1664 TPH						
Total Petroleum Hydrocarbons	ND	mg/L	3.1	0.92	1		06/08/16 17:37		
USGS I-3765 TSS	Analytical	Method: USG	S I-3765						
Total Suspended Solids	3.2	mg/L	1.0	1.0	1		06/06/16 13:13		
300.0 IC Anions 28 Days	Analytical	Method: EPA	300.0						
Sulfate	1030	mg/L	20.0	10.0	10		06/08/16 01:40	14808-79-8	



QUALITY CONTROL DATA

Project: NPDES-TB Wk1

Pace Project No.: 1267255

QC Batch: DUL/7297

QC Batch Method: **EPA 1664 TPH** Analysis Description:

Analysis Method:

EPA 1664 TPH

1664 SGT-HEM, TPH

Associated Lab Samples:

METHOD BLANK: 327080

1267255001

Associated Lab Samples: 1267255001

> Blank Reporting

Matrix: Water

Limit MDL Parameter Units Result Qualifiers Analyzed Total Petroleum Hydrocarbons ND 3.0 0.90 06/08/16 15:12 mg/L

LABORATORY CONTROL SAMPLE:

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Total Petroleum Hydrocarbons mg/L 20 13.4 67 64-132

MATRIX SPIKE SAMPLE: 327082

Date: 06/15/2016 04:51 PM

MS 1267347001 Spike MS % Rec Parameter Units Result Conc. Result % Rec Limits Qualifiers < 0.94 64-132 Total Petroleum Hydrocarbons 20.6 13.9 68 mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALITY CONTROL DATA

USGS I-3765

Project: NPDES-TB Wk1

Pace Project No.: 1267255

QC Batch: WET/24458

QC Batch Method: USGS I-3765 Analysis Description: USGS I-3765 Total Suspended Solids

Associated Lab Samples: 1267255001

METHOD BLANK: 325939 Matrix: Water

Associated Lab Samples: 1267255001

Blank Reporting
Parameter Units Result Limit MDL Analyzed Qualifiers

Analysis Method:

Total Suspended Solids mg/L ND 1.0 06/06/16 13:13

LABORATORY CONTROL SAMPLE: 325940

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers **Total Suspended Solids** mg/L 239 230 96 80-120

SAMPLE DUPLICATE: 325941

1267425001 Dup Max **RPD RPD** Parameter Units Result Result Qualifiers 312 10 Total Suspended Solids 344 10 mg/L

SAMPLE DUPLICATE: 325942

Date: 06/15/2016 04:51 PM

1267418001 Dup Max RPD RPD Parameter Units Result Result Qualifiers 40.0 Total Suspended Solids mg/L 40.0 0 10

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALITY CONTROL DATA

Project: NPDES-TB Wk1

Pace Project No.: 1267255

Date: 06/15/2016 04:51 PM

QC Batch: WETA/17138 Analysis Method: EPA 300.0

QC Batch Method: EPA 300.0 Analysis Description: 300.0 IC Anions

Associated Lab Samples: 1267255001

METHOD BLANK: 326333 Matrix: Water

Associated Lab Samples: 1267255001

ParameterUnitsBlank Reporting ResultReporting LimitMDLAnalyzedQualifiersSulfatemg/LND2.01.006/07/16 14:47

LABORATORY CONTROL SAMPLE: 326334

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Sulfate mg/L 50 52.0 104 90-110

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 326335 326336

MS MSD 1267072001 Spike Spike MS MSD MS MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits RPD RPD Qual Sulfate 20 mg/L 2.9 50 50 57.1 56.3 108 107 90-110

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 326337 326338

MS MSD 1267402008 MS MSD MS Spike Spike MSD % Rec Max Limits RPD Parameter Units Result Conc. Conc. Result Result % Rec % Rec RPD Qual Sulfate <1.0 50 50 51.9 52.3 102 103 90-110 1 20 mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALIFIERS

Project: NPDES-TB Wk1
Pace Project No.: 1267255

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

Date: 06/15/2016 04:51 PM

PASI-DUL Pace Analytical Services - Duluth
PASI-V Pace Analytical Services - Virginia



QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: NPDES-TB Wk1

Pace Project No.: 1267255

Date: 06/15/2016 04:51 PM

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
1267255001	SD 001 (Seep 020)	EPA 1664 TPH	DUL/7297		
1267255001	SD 001 (Seep 020)	USGS I-3765	WET/24458		
1267255001	SD 001 (Seep 020)	EPA 300.0	WETA/17138	3	

Requested Due Date Required Client Information:
Company: USS Corporation Vt. Iron, MN 55768 Company: USS Corporation
Address: P.O. Box 417 ITEM# SD 001 (Seep 020) Sample Ids must be unique One Character per box. (A-Z, 0-9 / , -) SAMPLE ID ADDITIONAL COMMENTS Fax MATRIX
Diniking Water
Water
Waste Water
Product
Soil/Soild
Oil
Wipe
Ar
Other Purchase Order#: Project Name: Copy To: Report To: Required Project Information: TS O A WA C E P WAT DE COM RELINQUISHED BY / AFFILIATION Š MATRIX CODE (see valid codes to left) Tom Moe 1 contraction SAMPLE TYPE (G=GRAB C=COMP) NPDES-TB Wk1 CO:11 9249 CO:17 9129 DATE START SAMPLER NAME AND SIGNATURE TIME COLLECTED SIGNATURE of SAMPLER: PRINT Name of SAMPLER: The Chain-of-Custody is a LEGAL DOCUM CHAIN-OF-CUSTODY / Analytical Request Document DATE END 6-1-16 DATE TIME SAMPLE TEMP AT COLLECTION 20,75 # OF CONTAINERS Invoice Information: Attention: Section C Pace Project Manager: Pace Profile #: Pace Quote: Address Company Name: Unpreserved H2SO4 an mark le HNO3 Preservatives alwarte. HCI NaOH ACCEPTED BY / AFFILIATION Na2S2O3 heather zika@pacelabs.com PM: MMW Methanol CLIENT: USS CORP MO# :1267255 Analyses Test Y/N TSS,SO4 DATE Signed: TRPH 1664 Due Date: 06/15/16 6-1-16/4-25 DATE <u>۷</u> TEMP in C Residual Chlorine (Y/N) Received on SAMPLE CONDITIONS y Agency lce (Y/N) Custody Sealed ζ ç Cooler (Y/N) Samples Intact (Y/N)

/Face Analytical*	

Document Name:

Sample Condition Upon Receipt Form

Document No : F-VM-C-001-Rev.09 Document Revised: 23Feb2 015

Page 1 of 1

Issuing Authority: Pace Virginia, Minnesota Quality Office

Sample Condition
Upon Receipt

ent Name:	Project

Ħ:

W0#:1267255

C1-10 USPS Fed Ex UPS Client Commercial Pace Other: Tracking Number: Optional: Proj. Due Date: Proj. Name: Custody Seal on Cooler/Box Present? Yes KNo Seals Intact? Yes **M**No Packing Material: Bubble Wrap Bubble Bags None Other: Huspad Temp Blank? Ares No Thermometer Used: 😿 140792808 ▲Samples on ice, cooling process has begun Type of Ice: Wet Blue None Biological Tissue Frozen? Yes No NA Cooler Temp Read °C: Cooler Temp Corrected °C: Temp should be above freezing to 6°C Correction Factor: +0, 3 Date and Initials of Person Examining Contents: 6/1//6 Chain of Custody Present? Yes No □N/A Chain of Custody Filled Out? Yes □No □N/A Chain of Custody Relinquished? **∑**Yes □No □n/a Sampler Name and Signature on COC? Yes □No □N/A Samples Arrived within Hold Time? ₹Yes □No □N/A Short Hold Time Analysis (<72 hr)? No. Yes □N/A Rush Turn Around Time Requested? Yes No □N/A Yes Sufficient Volume? No □N/A Correct Containers Used? Yes □No □N/A -Pace Containers Used? Yes □No □N/A Containers Intact? Yes □ No □N/A Filtered Volume Received for Dissolved Tests? **X**N/A Yes No 11. Note if sediment is visible in the dissolved containers. Sample Labels Match COC? Mes □No □N/A -Includes Date/Time/ID/Analysis Matrix:_ See pH log for results and additional preservation All containers needing acid/base preservation will be □Yes No documentation checked and documented in the pH logbook. Yes ☐ No **M**N/A Headspace in Methyl Mercury Container 13, Headspace in VOA Vials (>6mm)? □Yes **₽**N/A □No 14. Trip Blank Present? Yes □No **₩**N/A 15. Trip Blank Custody Seals Present? N/A []]Yes No Pace Trip Blank Lot # (if purchased): CLIENT NOTIFICATION/RESOLUTION Field Data Required? Yes No Person Contacted: Comments/Resolution;

FECALWAIVER ON FILE Y	F	E	CAL	WAI	VER	ON	FILE	Υ
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TEMPERATURE WAIVER ON FILE

Project Manager Review:

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office 928 61 95

hold, incorrect preservative, out of temp, incorrect containers)

Intra-Regional Chain of Custody



Workorder: 1267255	Workorder Name: NPDES-TB Wk1	0	Owner Received	r Received Date: 6/1/2016	Due Date: 6/15/2016
Received at:	Send To Lab:			Requested Analysis	nalysis
Pace Analytical Virginia	Pace Analytical Duluth				
315 Chestnut Street	4730 Oneota Street				
Virginia, MN 55792	Duluth, MN 55807				
1 1010 (210) 172-1072	(11016 (110) 11, 0000				
			I TPH		
Melisa M Woods		Preserved Containe	ontainers PA 1864		
Ifem Sample ID	Sample Collect Type Date/Time Lab D Mr	Matrix HC			LAB USE ONLY
1 SD 001 (Seep 020)	PS 6/1/2016 11:00 1267255001 W	Water 3	×		
2					
ω					
4					`
5					
本者の大学の表示ではないののでは、「一年の大学の大学の大学の大学の大学の大学の大学の大学の大学の大学の大学の大学の大学の					Comments
Transfers Released By	Date/Time Received By		Date/Time		
1 -2-	(dzh. 1250 Ravena	Rovenaderia	6/2110 1050		
2		•			
3					
4					
Cooler Temperature on Receipt	Receipt $O.Q$ °C Custody Seal (Y) or	Z	Received on Ice	Y) or N	Samples Intact or N

^{***}In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document. This chain of custody is considered complete as is since this information is available in the owner laboratory.

Pace Analytical *

Document Name: Sample Condition Upon Receipt Form

Document No.: F-DUL-C-001-Rev.01 Document Revised: 22Jan2016 Page 1 of 1

Issuing Authority: Pace Virginia, Minnesota Quality Office

ample Condition Client Name;		ſ	Project #:	
Upon Receipt				
Courier: Fed Ex UPS	USPS	CI		
☐Commercial ☐Pace	🔀 Other:_	Mik	<u> </u>	
racking Number:				
ustody Seal on Cooler/Box Present? XYes 🗌	No	Seals In	tact? 🌾	Yes No Optional: Proj. Due Date: Proj. Name:
acking Material: 🔲 Bubble Wrap 🗡 Bubble Baj	gs 🔲 No	one [Other:	Temp Blank? Yes No
ermometer Used: 🖳 800051	Type of I	ce: 📐	Wet _	Blue None Samples on ice, cooling process has begu
Cooler Temp Read °C: O Cooler Temp Comp should be above freezing to 6°C Correction Fact				Biological Tissue Frozen? Yes No KN. I Initials of Person Examining Contents: Comments:
Chain of Custody Present?	Yes	□No	□n/a	1.
Chain of Custody Filled Out?	Yes	□No	□N/A	2.
Chain of Custody Relinquished?	Yes	□No	□n/a	3.
Sampler Name and Signature on COC?	∐Yes	□No	₽ N/A	4.
Samples Arrived within Hold Time?	Yes	□No	□n/a	5.
Short Hold Time Analysis (<72 hr)?	Yes	X No	□n/a	6.
Rush Turn Around Time Requested?	□Yes	₩No	□n/a	7.
Sufficient Volume?	Yes	□No	□n/a	8.
Correct Containers Used?	¥Yes	□No	□n/a	9.
-Pace Containers Used?	₹¥Yes	□No	□N/A	
Containers Intact?	₹Yes	□No	□N/A	10.
Filtered Volume Received for Dissolved Tests?	Yes	□No	Æ N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	⋉ Yes	□No	□N/A	12.
-Includes Date/Time/ID/Analysis Matrix: 125				
All containers needing acid/base preservation will be checked and documented in the pH logbook.	☐ Yes	∏No	∏N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	∐Yes	□No	[]N/A	13.
Headspace in VOA Vials (>6mm)?	□Yes	No	[]N/A	14.
Trip Blank Present?	∐Yes	□No	□n/a	15.
Trip Blank Custody Seals Present?	Yes	□No	∐ N/A	
Pace Trip Blank Lot # (if purchased):	***			
CLIENT NOTIFICATION/RESOLUTION				Field Data Required? Yes No
Person Contacted:				Date/Time:
Comments/Resolution:				

FECAL WAIVER ON FILE

TEMPERATURE WAIVER ON FILE

Project Manager Review: Date: 6-3-16

lote: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of

old, incorrect preservative, out of temp, incorrect containers)